



## Diamond structure recovery during ion irradiation at elevated temperatures



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### ABSTRACT

CVD diamond is irradiated by 5 MeV carbon ions, with each sample held at a different temperature (300–873 K) during irradiations. The defect structures resulting from the irradiations are evident as vacancy, interstitial and amorphous carbon signals in Raman spectra. The observed variation of the full width at half maximum (FWHM) and peak position of the diamond peak suggests that disorder in the diamond lattice is reduced for high temperature irradiations. The dumbbell interstitial signal is reduced for irradiations at 873 K, which suggests this defect is unstable at these temperatures and that interstitials have migrated to crystal surfaces. Near edge X-ray absorption fine structure (NEXAFS) spectroscopy results indicate that damage to the diamond structure at the surface has occurred for room temperature irradiations, however, this structure is at least partially recovered for irradiations performed at 473 K and above. The results suggest that, in a high temperature irradiation environment such as a nuclear fusion device, *in situ* annealing of radiation-created defects can maintain the diamond structure and prolong the lifetime of diamond components.

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### 1. Introduction

Diamond is the carbon allotrope that offers the best resistance to radiation damage due to the strong binding energy of its lattice. Due to CVD diamond's versatility for applications in extreme radiation environments it has been proposed for use in nuclear fusion devices as windows through which heating power can be delivered [1], as radiation detectors [2,3], and as a protective coating on high ion flux components such as the divertor [4]. The diamond material of these fusion components will be exposed to irradiation with energetic particles, including 14 MeV neutrons produced by deuterium–tritium (D–T) reactions. Due to the cost and time required to reach appreciable doses of relevant neutron irradiations, a rational choice to simulate the damage caused by the 14 MeV neutrons from the D–T fusion reaction is that of the most damaging knock-on atoms, i.e. MeV carbon atoms. High energy particle bombardment of diamond produces damage in the form of vacancies and interstitial atoms. With enough damage, the diamond structure can be lost and with annealing, the damaged region will

graphitise [5,6]. In a nuclear fusion environment, materials will potentially have a temperature of several hundred Kelvin, and damage due to the neutron flux is expected to be of the order of 1 displacement per atom (dpa) [7], hence there is a need to investigate the irradiation of diamond at elevated temperatures.

There have been several decades of investigation into the effect of sample temperature during irradiation of diamond with high energy particles, much of which is summarised in a review by Prins [8] and the references therein. The motivation of much of this earlier work was the use of ion implantation for doping of diamond [9,10], and diamond growth [11,12]. Elevated sample temperatures during implantation were found to drive interstitials to the surface, but resulted in agglomeration of vacancies in extended defects [12]. These extended defects were not ideal for doping applications, and the temperature-enhanced mobility meant the dopants could also diffuse from their desired location [13]. The technique of cold implantation followed by rapid anneal was instead adopted [14], and much of the work in literature henceforth has applied this technique. Prawer and Kalish found the activation energy for defect diffusion in the ion track thermal spike during hot implants to be  $\sim 0.2$  eV [15], whereas post-implantation annealing experiments found a value for C interstitial diffusion to be 1.3 eV [16].

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Kalish et al. used Raman spectroscopy of post-implantation annealed diamond to monitor the dynamics of defects, and showed that above 900 K a diamond dumbbell interstitial peak at  $1630\text{ cm}^{-1}$  disappeared, whereas beyond 1100 K a vacancy-related peak at  $1490\text{ cm}^{-1}$  reduced in intensity [17]. Similar Raman results were found by Orwa et al., who also used polarisation of the laser light to show a broad skewed peak centred at  $1245\text{ cm}^{-1}$  that was assigned to amorphous carbon [18]. In irradiated polycrystalline films, a luminescent band ascribed to the H3 centre was observed around  $1250\text{ cm}^{-1}$  [19].

Partial electron yield (PEY) Near-edge X-ray absorption fine structure (NEXAFS) spectroscopy is a surface sensitive (typically less than 5 nm) synchrotron-based technique that shows the local bonding environment of the absorbing element. Previously, it was demonstrated that NEXAFS is sensitive to formation of defective structure in diamond bombarded with 30 keV Xe ions [20]. For fusion applications, NEXAFS has been used to investigate carbon ion irradiation of diamond [21], and exposure of diamond to hydrogen plasma [22]. NEXAFS studies also showed that surface termination can effect core-level bulk excitons [23], and diamond graphitised by implantation can be partially recovered by post-implantation annealing [24].

An understanding of the damage resulting from irradiation at different temperatures is of interest for diamond applications in nuclear fusion devices, to aid the understanding of component failure and lifetimes. In this work we use Raman and NEXAFS spectroscopy to investigate the damage structures arising in diamond as a result of high energy ion irradiation at a range of temperatures between 300 K and 873 K.

## 2. Experimental

Chemical vapour deposited polycrystalline diamond of grade TM-100 was obtained from Element 6 Ltd. The diamond samples were free standing  $10\text{ mm} \times 10\text{ mm} \times 0.25\text{ mm}$  plates and were used as-received. Samples were irradiated with 5 MeV  $\text{C}^{2+}$  ions, using a 5  $\mu\text{A}$  broad beam, in a system pumped with oil-free vacuum pumps and operating vacuum of  $<1 \times 10^{-7}$  Torr. Each sample was irradiated to a fluence of  $1 \times 10^{17}$  ions  $\text{cm}^{-2}$ , which was monitored by integrating the drain current from the sample. Sample temperature was monitored using a thermocouple attached to the sample holder, which was interfaced with the sample heater. Different samples were irradiated at 300 K, 473 K, 673 K and 873 K. The sample irradiated at 300 K exhibited a temperature rise of approximately 20 K throughout the irradiation, which is considered to be negligible for the purposes of this experiment. SRIM estimates of the range and damage profiles for 5 MeV  $\text{C}^{2+}$  irradiation of diamond have previously been reported [21]. The predicted range was 2.4  $\mu\text{m}$ . The predicted damage created by  $1 \times 10^{17}$  ions  $\text{cm}^{-2}$  is 1 dpa, averaged over the range, assuming displacement energy of 50 eV.

Raman spectra were collected using a Renishaw inVia Raman spectrometer equipped with an Argon ion laser (514 nm) and a Peltier cooled CCD detector. Stokes shifted Raman spectra were collected in the range of  $\sim 100\text{--}2000\text{ cm}^{-1}$  with a spectral resolution of  $\sim 1.7\text{ cm}^{-1}$  for the 1800 lines  $\text{mm}^{-1}$  grating that was used. The spot size was approximately 1.5  $\mu\text{m}$  for 50 $\times$  magnification. The probing depth for this wavelength in diamond is of the order of a few  $\mu\text{m}$ . Six spectra were collected from different locations near the centre of each sample. Each spectra was background subtracted by fitting a polynomial to the boundary of the region of interest, before fitting or averaging. Peaks were fit using a Voigt function, with constraints to the peak positions on the order of  $\pm 25\text{ cm}^{-1}$ .

Near edge X-ray absorption fine structure (NEXAFS) spectroscopy measurements were performed at the Soft X-ray Spectroscopy beamline at the Australian Synchrotron [25]. Details of the experimental setup and normalisation process can be found elsewhere [22]. An incident photon energy range of 270–320 eV was used corresponding to the carbon K-edge. Samples were mounted at an angle of  $45^\circ$  to the incident X-ray beam. The NEXAFS signal was collected in partial electron yield (PEY) mode with the cut-off voltage set to 135.5 V. The mean free paths of electrons at this energy are of the order of a few nm, resulting in a highly surface sensitive measurement. Carbon K-edge NEXAFS peak assignments are taken from Guenette et al. and the references therein [22]. The photon energy calibration and normalisation process was carried out as per the methods outlined by Watts et al. [26,27]. The  $\text{sp}^2$  fraction of the surface (<5 nm depth) was calculated by applying the well-established method used for electron energy loss spectroscopy (EELS) measurements [28] to the NEXAFS data, details of which can be found elsewhere [21].

## 3. Results

Fig. 1 shows Raman spectra of diamond irradiated at different temperatures and an unirradiated sample. Each spectrum has been background subtracted, normalised to the height of the diamond peak, and the figure shows the averaged spectrum for each set of irradiation conditions. The spectrum of the unirradiated sample exhibits a sharp diamond peak at  $1333\text{ cm}^{-1}$ , and a broad disordered carbon peak around  $1500\text{ cm}^{-1}$ . After irradiation peaks are evident at  $1630\text{ cm}^{-1}$ , which are assigned to a carbon interstitial diamond dumbbell defect. Vacancy-related peaks are evident at  $1490\text{ cm}^{-1}$  and  $1420\text{ cm}^{-1}$  [17]. There is broadening of the diamond peak about  $1333\text{ cm}^{-1}$  following irradiation, which indicates variations to the diamond bonding state that are introduced by a distribution of defective diamond bonds. For wavenumbers less than the  $1333\text{ cm}^{-1}$  diamond peak there appears to be either an asymmetric broadening of the diamond peak, indicative of diamond bonds that are distorted by the presence of nearby defects, or this signal may originate from a defect state such as the H3 centre [19]. The broad signal of disordered carbon around  $1500\text{ cm}^{-1}$  appears to be greatest for the sample irradiated at 300 K, but with increasing irradiation temperature it decreases to be approximately half of this intensity for the samples irradiated at 673 K and 873 K. The Raman spectra of the diamond irradiated at 300 K and 473 K exhibit the greatest differences when compared to that

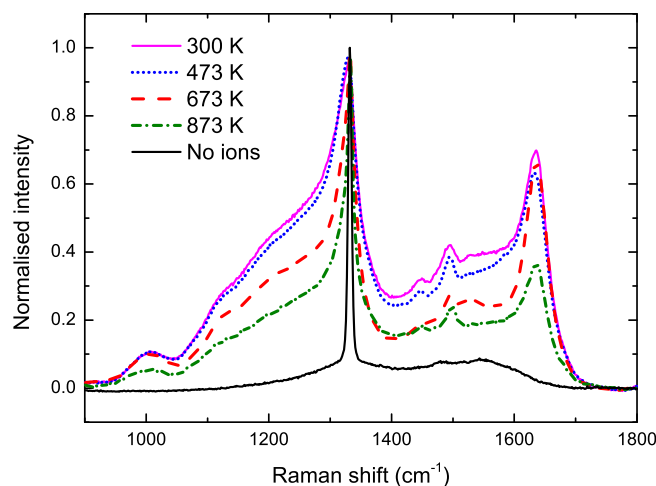


Fig. 1. Raman spectra of diamond irradiated at different temperatures.

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